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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
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10/579,176

10/24/2007

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P2003J095

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7590

02/11/2009

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EXAMINER

ROBINSON, RENEE E

ART UNIT

PAPER NUMBER

1797

MAIL DATE

DELIVERY MODE

02/11/2009

PAPER

**Please find below and/or attached an Office communication concerning this application or proceeding.**

The time period for reply, if any, is set in the attached communication.

<b>Office Action Summary</b>	<b>Application No.</b> 10/579,176	<b>Applicant(s)</b> GREANEY ET AL.	
	<b>Examiner</b> RENEE ROBINSON	<b>Art Unit</b> 1797	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

### Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

### Status

- 1) ☒ Responsive to communication(s) filed on 15 May 2006.
- 2a) ☐ This action is **FINAL**.                      2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

### Disposition of Claims

- 4) ☒ Claim(s) 1-40 is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 1-40 is/are rejected.
- 7) ☐ Claim(s) \_\_\_\_\_ is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_\_ are subject to restriction and/or election requirement.

### Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☒ The drawing(s) filed on 15 May 2006 is/are: a) ☒ accepted or b) ☐ objected to by the Examiner.  
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).  
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

### Priority under 35 U.S.C. § 119

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All    b) ☐ Some \*    c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
  2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.
  3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

### Attachment(s)

- |  |   |
|--|---|
| 1) <input checked="" type="checkbox"/> Notice of References Cited (PTO-892)                                | 4) <input type="checkbox"/> Interview Summary (PTO-413)<br>Paper No(s)/Mail Date. _____ |
| 2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948)                       | 5) <input type="checkbox"/> Notice of Informal Patent Application                       |
| 3) <input type="checkbox"/> Information Disclosure Statement(s) (PTO/SB/08)<br>Paper No(s)/Mail Date _____ | 6) <input type="checkbox"/> Other: _____  |

## **DETAILED ACTION**

### ***Claim Rejections - 35 USC § 112***

1. The following is a quotation of the second paragraph of 35 U.S.C. 112:  
  
The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.
2. Claims 19-21 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.
3. Regarding claim 19, it is unclear what is meant by the phrase "any means known". It is unclear whether the claim scope would be the same were it not to be present or whether it has some other meaning, including perhaps an invocation of "means or step plus function" claim language, as provided for by 35 USC § 112, sixth paragraph. If the latter, "means for" language is suggested. Moreover, if so, it is unclear whether clear linkage to disclosed means has been provided in the Specification.

### ***Double Patenting***

1. The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory obviousness-type double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., *In re Berg*, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); *In re Goodman*, 11 F.3d 1046, 29

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USPQ2d 2010 (Fed. Cir. 1993); *In re Longi*, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); *In re Van Ornum*, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); *In re Vogel*, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); and *In re Thorington*, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

2. A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on a nonstatutory double patenting ground provided the conflicting application or patent either is shown to be commonly owned with this application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement.

3. Effective January 1, 1994, a registered attorney or agent of record may sign a terminal disclaimer. A terminal disclaimer signed by the assignee must fully comply with 37 CFR 3.73(b).

**4. Claims 1-9, 12-15, 18-20, 22-29, 33, 35, 36 and 38 are provisionally rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1, 2 and 4-13 of copending Application No. 10/580,582 in view of Plummer et al (U.S. Patent 3,487,012).**

5. Regarding claims 1-3, 6, 12-14, 22, 24, 27 and 33 of the instant application and claims 1, 2 and 13 of the copending application, both claim a hydrotreating/hydroprocessing of a hydrocarbon feedstream containing nitrogen contaminants, comprising:

- a) providing a sulfuric acid solution having a sulfuric acid concentration of at least about 75%, based on the sulfuric acid solution;
- b) contacting the feedstream with the sulfuric acid solution under conditions effective at removing at least 60 wt.% (or about 80 wt.%) of the nitrogen compounds contained in the feedstream, wherein the volumetric treat rate of the sulfuric acid solution is greater than about 0.5 vol.%, based on the feedstream; and
- c) processing the hydrocarbon effluent by a hydrotreating process.

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6. The differences between the instant application and the copending application are:

- the instant application claims a diesel boiling range feedstream, whereas the copending application claims a lube oil boiling range feedstream;
- the instant application claims contacting the sulfuric acid and the feedstream by a non-dispersive method, whereas the copending application does not. However, the copending application claims contacting by a non-dispersive method in dependent claim 7; and
- the instant application claims hydrotreating with hydrotreating catalysts containing at least one Group VI metal oxide and at least one Group VIII metal oxide under conditions effective at removing or converting at least a portion of the sulfur contained in the product.

7. Plummer discloses a method for the hydroprocessing of a nitrogen-containing diesel boiling range (400-750°F) feedstream (column 2 lines 21-28). The process comprises contacting a nitrogen-containing diesel boiling range feedstream with a sulfuric acid solution having a concentration greater than 85% (column 3, lines 21-25). Plummer further discloses hydrotreating the diesel boiling range hydrocarbons with metal oxides of Groups VI-B and VIII of the Periodic Table (column 3, lines 13-15).

8. Therefore, a person of ordinary skill in the art would have been motivated to modify the process as claimed in the copending application to hydroprocess feedstreams with boiling points outside of the lube oil boiling range and further to hydrotreat with metal oxide catalysts from Groups IV-B and VIII, as suggested by

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Plummer. One having ordinary skill in the art would have been motivated to do this in order to effectively remove nitrogen from feedstreams with boiling points outside of the diesel boiling range.

9. Regarding claims 4 and 25 of the instant application and claim 4 of the copending application, both claim a nitrogen content in the feedstream in the range of 50-1000 or 75-800 wpppm.

10. Regarding claims 5 and 26 of the instant application and claim 5 of the copending application, the copending application claims that the nitrogen in the feedstock is basic and non-basic heterocyclic nitrogen compounds, whereas the instant application claims carbazole and/or substituted carbazoles. However, carbazole is a type of heterocyclic nitrogen compound.

11. Claims 7 and 28 of the instant application and claim 6 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

12. Claim 8 of the instant application and claim 10 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

13. Regarding claims 9 and 29 of the instant application and claim 11 of the copending application, the copending application claims adding a diluent to the sulfuric acid solution to adjust the concentration, whereas the instant application claims adding water to adjust the sulfuric acid concentration. However, water is a type of diluent.

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14. Claim 15 of the instant application and claim 7 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

15. Claims 18, 19 and 35 of the instant application and claim 8 of the copending application all pertain to separating the sulfuric acid solution from the effluent. Claim 8 of the copending application differs from claims 18 and 35 of the instant application in that the instant application does not claim separating "by any means known to be effective". Nevertheless, separating by "any means known to be effective" accomplishes the claim scope of separating the two streams. Claim 19 of the instant application and claim 8 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

16. Claims 20 and 36 of the instant application and claim 9 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

17. Claims 23 and 38 of the instant application and claim 12 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

18. This is a provisional obviousness-type double patenting rejection.

**19. Claims 1-9, 11-15, 18-20, 22-29, 31-33, 35, 36 and 38 are provisionally rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1, 2, 5-16, 18 and 19 of copending Application No. 10/579,178 in view of Plummer et al (U.S. Patent 3,487,012).**

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20. Regarding claims 1-3, 6, 13, 22, 24 and 27 of the instant application and claims 1, 2, 7 and 18 the copending application, both claim a hydroprocessing/hydrotreating of hydrocarbon feedstream containing nitrogen contaminants, comprising:

- a) providing a sulfuric acid solution having a sulfuric acid concentration of at least about 75%, based on the sulfuric acid solution;
- b) contacting the feedstream with the sulfuric acid solution under conditions effective at removing at least 60 wt.% of the nitrogen compounds contained in the feedstream, wherein the volumetric treat rate of the sulfuric acid solution is greater than about 0.5 vol.%, based on the feedstream; and
- c) processing the hydrocarbon effluent by a process selected from hydrotreating, hydrocracking, hydrodewaxing, and hydrofinishing.

21. The differences between the instant application and the copending application are:

- the copending application claims a broad feedstream of hydrocarbons for the hydroprocessing method, while the instant application claims a diesel boiling range feedstream. However, the copending application claims that the hydrocarbon feedstream boils above about 300°F in dependent claim 2, which encompasses diesel boiling range feedstreams;
- The instant application claims non-dispersive contacting methods. However, the copending application claims non-dispersive contacting methods in claim 14;



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- the instant application claims hydrotreating with hydrotreating catalysts containing at least one Group VI metal oxide and at least one Group VIII metal oxide under conditions effective at removing or converting at least a portion of the sulfur contained in the product, whereas the copending application claims contacting a hydrocarbon stream with a hydroprocessing catalyst in a reaction stage in dependent claim 18.

22. Plummer discloses a method for the hydroprocessing of a nitrogen-containing diesel boiling range (400-750°F) feedstream (column 2 lines 21-28). The process hydrotreating the diesel boiling range hydrocarbons with metal oxides of Groups VI-B and VIII of the Periodic Table (column 3, lines 13-15).

23. Therefore, a person of ordinary skill in the art would have been motivated to modify the process as claimed in the copending application to hydroprocess with metal oxide catalysts from Groups IV-B and VIII, as suggested by Plummer. One having ordinary skill in the art would have been motivated to do this in order to effectively hydrotreat the feedstream to produce a product with a reduced level of sulfur and nitrogen contaminants.

24. Regarding claims 4 and 25 of the instant application and claim 5 of the copending application, the copending application claims a feedstream containing about 25-2500wppm nitrogen, which encompasses the instant claimed nitrogen content of 50-1000wppm and 75-800wppm, respectively.

25. Claims 5 and 26 of the instant application and claim 6 of the copending application both claim carbazole and/or substituted carbazoles.

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26. Claims 7 and 28 of the instant application and claim 8 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

27. Claim 8 of the instant application and claim 9 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

28. Regarding claims 9 and 29 of the instant application and claim 10 of the copending application, the copending application claims adding a diluent to the sulfuric acid solution to adjust the concentration, whereas the instant application claims adding water to adjust the sulfuric acid concentration. However, water is commonly used in the art as a diluent.

29. Regarding claims 11 and 31 of the instant application and claim 11 of the copending application, both claim a sulfur concentration in the product stream(s) of about 0.1 to about 25 wt. % less than the feedstream.

30. Regarding claims 12 and 32 of the instant application and claim 12 of the copending application, both claim a yield loss within the range of about 0.5 to about 6 wt.%.

31. Claims 14 and 33 of the instant application and claim 13 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

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32. Claim 15 of the instant application and claim 14 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

33. Claims 18 and 35 of the instant application claims separating the product from the sulfuric acid solution, which is claimed in step c) of independent claim 1 of the copending application.

34. Claim 19 of the instant application and claim 15 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

35. Claims 20 and 36 of the instant application and claim 16 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

36. Claims 23 and 38 of the instant application and claim 19 of the copending application are identical except with regards to the differences in the independent claims, as discussed above.

37. This is a provisional obviousness-type double patenting rejection.

### ***Claim Rejections - 35 USC § 102***

38. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless –

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

**39. Claims 1-6, 10, 11, 13-15, 17 and 23 are rejected under 35 U.S.C. 102(b) as being anticipated by Skomoroski et al (U.S. Patent 3,123,550).**

40. Regarding claims 1-3, 6, 13 and 14, Skomoroski discloses a method for hydrotreating a shale oil distillate feedstream containing both nitrogen and sulfur contaminants (column 1, lines 10-17; Column 2, Table 1). The process comprises:

- a) Providing a sulfuric acid solution having a sulfuric acid concentration of at least about 75 wt.% (96.6 wt.%), based on the sulfuric acid solution (column 3, lines 54-58);
- b) Contacting the feedstream containing both nitrogen and sulfur heteroatoms with the sulfuric acid solution under conditions effected at removing at least about 85 wt.% (over 90%) of the nitrogen compounds contained in the feedstream, thereby producing at least a product and a used sulfuric acid solution, wherein the volumetric treat rate of the sulfuric acid solution is greater than about 0.5 vol.% based on the feedstream (column 3, lines 53-67; Fig. 1); and
- c) Hydrotreating the diesel boiling range product. (column 3, lines 59-61).

41. Skomoroski discloses that the shale oil distillate feedstream boils in the range of 350-1000°F (column 2, line 46), encompassing a diesel boiling range feedstream as defined in the instant application.

42. Regarding claim 4, Skomoroski discloses that the feedstream contains 0.5-4.0 wt.% nitrogen (500-4000 wppm), which encompasses the claimed range of 50-1000 wppm nitrogen (column 2, line 49).

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43. Regarding claim 5, Skomoroski discloses that the nitrogen present in the feedstream includes aromatic nitrogen compounds. Skomoroski does not expressly disclose that the feedstream contains carbazole and/or substituted carbazole. However, as evidenced by Min et al ("Carbazole-type compounds in crude oils"), nitrogen-containing species in petroleum consist of carbazole-type compounds, such as carbazoles and their derivatives (benzocarbazoles, dibenzocarbazoles and their alkylated derivatives) (Chinese Science Bulletin, Vol. 43 No. 8, p. 669). Therefore, the feedstream disclosed by Skomoroski would inherently contain carbazole and/or substituted carbazole compounds.

44. Regarding claim 10, Skomoroski discloses an example in which the feedstream initially contained 2.07 wt.% nitrogen (2070 wppm) and the process removed over 90% of the nitrogenous contaminants, corresponding to a final concentration of nitrogen in the product stream of less than 200 wppm (column 2, lines 54-55 and 67-68).

45. Regarding claim 11, Skomoroski discloses that the sulfur content of a feedstock with 0.4 wt.% sulfur is reduced to a negligible amount, corresponding to a product with a sulfur content that is at least 0.1 wt.% to approximately 0.4 wt.% less than the feedstream (column 4, lines 11 and 16-17).

46. Regarding claims 15 and 17, Skomoroski discloses that the feedstream and sulfuric acid solution are contacted by a dispersive method which includes stirring the mixture in a mixing tank (column 1, lines 68-71).

***Claim Rejections - 35 USC § 103***

47. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

48. The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

49. This application currently names joint inventors. In considering patentability of the claims under 35 U.S.C. 103(a), the examiner presumes that the subject matter of the various claims was commonly owned at the time any inventions covered therein were made absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and invention dates of each claim that was not commonly owned at the time a later invention was made in order for the examiner to consider the applicability of 35 U.S.C. 103(c) and potential 35 U.S.C. 102(e), (f) or (g) prior art under 35 U.S.C. 103(a).

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**50. Claims 7 and 12 are rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Lockwood et al (U.S. Patent 3,749,666).**

51. Skomoroski is relied upon as set forth above in the rejection of claims 1, 2 and 10.

52. Regarding claim 7, Skomoroski does not expressly disclose that the sulfuric acid solution is obtained from an alkylation process unit.

53. Lockwood discloses a method for the improvement of petroleum distillate comprising contacting (emulsifying) the petroleum stream with spent alkylation acid (column 2, lines 2-8). Lockwood further discloses that while fresh sulfuric acid can be used, there is an economic advantage to using spent alkylation acid (column 2, lines 45-48).

54. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by using spent sulfuric acid from an alkylation process unit in order to reduce the amount of fresh sulfuric acid required for the process.

55. Regarding claim 12, Skomoroski does not expressly disclose that the yield loss attributed to the sulfuric acid treatment is about 0.5 to about 6 wt.%.

56. Lockwood discloses that controlled acidity minimizes oil loss from the petroleum distillate feed (column 3, lines 7-9). Therefore, it would have been obvious to a person of ordinary skill in the art at the time the invention was made to control the acidity of the

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acid solution and hydrocarbon feedstream contacting stage in order to minimize loss associated with the sulfuric acid.

**57. Claims 8 and 9 are rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Lockwood et al (U.S. Patent 3,749,666) and in further view of Parvinen et al (U.S. Patent 6,007,722).**

58. Skomoroski in view of Lockwood is relied upon as set forth above in the rejection of claim 7.

59. Regarding claim 8, Skomoroski in view of Lockwood does not expressly disclose that the alkylation process comprises:

- a) Combining an olefinic hydrocarbon feedstream containing C<sub>4</sub> olefins with isobutane to form a hydrocarbonaceous mixture; and
- b) Contacting the hydrocarbonaceous mixture with sulfuric acid under conditions effective for producing at least an alkylate and a sulfuric acid solution having an acid concentration of at least about 75 wt. %.

60. Parvinen discloses a process for recycling and reusing spent sulfuric acid obtained from an alkylation process (column 1, lines 60-64; column 2, lines 23-25). The alkylation process comprises:

- a) Combining an olefinic hydrocarbon feedstream containing C<sub>4</sub> olefins (butene) with isobutane to form a hydrocarbonaceous mixture; and
- b) Contacting the hydrocarbonaceous mixture with sulfuric acid under conditions effective for producing at least an alkylate and a sulfuric acid



solution having an acid concentration of at least about 75 wt. % (85-88%)  
(column 1, lines 57-64; column 2, lines 5-7).

61. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski in view of Lockwood by obtaining the sulfuric acid from an alkylation process as described by Parvinen.

62. One having ordinary skill in the art would have been motivated to do this in order to produce alkylates and efficiently use sulfuric acid for multiple processes.

63. Regarding claim 9, Lockwood discloses that the alkylation acid contains water as obtained from the alkylation unit (column 2, lines 40-42). Lockwood further discloses that the acidity should be controlled so as to minimize loss of the petroleum distillate (column 3, lines 7-9).

64. Therefore, it would have been obvious to a person of ordinary skill in the art at the time the invention was made to use water to adjust to sulfuric acid concentration of the acid solution in order to minimize the loss of hydrocarbon feed.

**65. Claim 16 is rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Kenyon (U.S. Patent 3,758,404).**

66. Skomoroski is relied upon as set forth above in the rejection of claims 1 and 15.

67. Kenyon discloses a process for liquid-liquid mass transfer comprising contacting a hydrocarbon feedstream with a sulfuric acid solution through the use of a fiber film contactor, a non-dispersive contacting method (column 2, lines 20-30; column 4, lines 37-40). Kenyon further discloses that the interface between the two liquids is constantly

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changed, thereby increasing the rate of transfer of a component between two liquids (column 2, lines 50-53).

68. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by contacting the sulfuric acid solution and hydrocarbon feedstream by using a fiber film contactor. One would have been motivated to do this because fiber film contactors promote efficient mass transfer between two contacting liquids.

**69. Claims 18, 19, 22 and 23 are rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Plummer et al (U.S. Patent 3,487,012).**

70. Skomoroski is relied upon as set forth above in the rejection of claim 1.

71. Regarding claims 18 and 19, Skomoroski does not expressly disclose separating the sulfuric acid solution from the product.

72. Plummer discloses a process for improving the properties of aromatic concentrates comprising contacting the concentrate with a sulfuric acid solution and subsequently separating the acid solution from the product (column 3, lines 30-31). Plummer states that the sulfuric acid is substantially removed from the feed material, indicating that the separation technique was effective in separating an acid from a hydrocarbon stream.

73. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by separating

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the sulfuric acid from the product in order to provide a product which is substantially free of the acid solution.

74. Regarding claim 22, Skomoroski discloses hydrotreating the acid treated product with cobalt and nickel (Group VIII metals) under conditions effective in reducing the sulfur content of the product, but does not expressly disclose contacting the product with a hydrotreating catalyst containing at least one Group VI metal oxide and at least one Group VIII metal oxide.

75. Plummer discloses hydrotreating the acid treated product with a catalyst containing Groups VI-B and VIII metal oxides (column 3, lines 13-20). Plummer further discloses that the hydrotreatment is effective in improving properties, such as color and odor, of the aromatic concentrates (column 2, lines 44-45).

76. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by hydrotreating the acid treated product with a catalyst containing Group VI and VIII metal oxides in order to effectively remove sulfur contaminants and provide a higher quality product.

77. Regarding claim 23, Skomoroski discloses contacting the product with caustic and water; however, this step occurs after hydrotreatment (column 2, lines 12-21).

78. Plummer discloses contacting the product with caustic prior to hydrotreating. The caustic solution is added in an amount sufficient to neutralize the acid values remaining in the product, or in other words, reduce the total acid number (column 3, lines 32-35; column 6, lines 22-24).

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79. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by doing the caustic contacting stage prior to hydrotreatment in order to neutralize the hydrocarbon product and more readily prepare it for hydrotreatment.

**80. Claims 20 and 21 are rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Plummer et al (U.S. Patent 3,487,012) and in further view of Norman (U.S. Patent 4,432,865).**

81. Skomoroski in view of Plummer is relied upon as set forth above in the rejection of claims 18 and 19.

82. Regarding claims 20 and 21, Skomoroski in view of Plummer does not expressly disclose the separation device used to separate the sulfuric acid solution from the product.

83. Norman discloses a process for treating used motor oil or synthetic crude oil with a mineral acid, such as sulfuric acid, for removing undesired nitrogen-containing materials (column 1, lines 18-21; column 8, line 3). Norman discloses that the acid solution is separated from the oil product by high speed centrifuges or clay or cellulose fiber films and the residue from the separation contains the metal contaminants and acid solution (component A) (column 14, lines 11-18).

84. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski in view of Plummer by using high speed centrifuges or fiber films to separate the sulfuric acid

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solution from the product in order to effectively remove the acid solution and contaminants from the desired product.

**85. Claims 24-27, 30, 31, 33-35 and 38 are rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Plummer et al (U.S. Patent 3,487,012) and in view of Kenyon (U.S. Patent 3,758,404).**

86. Regarding claims 24, 33 and 34, Skomoroski discloses a method for hydrotreating a shale oil distillate feedstream containing both nitrogen and sulfur contaminants (column 1, lines 10-17; Column 2, Table 1). The process comprises:

- a) Providing a sulfuric acid solution having a sulfuric acid concentration of at least about 75 wt.% (96.6 wt.%), based on the sulfuric acid solution (column 3, lines 54-58);
- b) Contacting the feedstream containing both nitrogen and sulfur heteroatoms with the sulfuric acid solution under conditions effected at removing at least about 85 wt.% (over 90%) of the nitrogen compounds contained in the feedstream, thereby producing at least a product and a used sulfuric acid solution, wherein the volumetric treat rate of the sulfuric acid solution is greater than about 0.5 vol.% based on the feedstream (column 3, lines 53-67; Fig. 1); and
- c) Hydrotreating the diesel boiling range product with a hydrotreating catalyst containing cobalt or nickel (Group VI metals). (column 3, lines 20-24 and 59-61).

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87. Skomoroski discloses that the shale oil distillate feedstream boils in the range of 350-1000°F (column 2, line 46), encompassing a diesel boiling range feedstream boiling in the range as claimed.

88. Skomoroski does not expressly disclose:

- b) Contacting the sulfuric acid solution and oil feedstream through the use of a non-dispersive method;
- c) A hydrotreating catalyst containing at least one Group VI metal oxide and at least one Group VIII metal oxide.

89. Kenyon discloses a process for liquid-liquid mass transfer comprising:

- b) Contacting a hydrocarbon feedstream with a sulfuric acid solution through the use of a fiber film contactor, a non-dispersive contacting method (column 2, lines 20-30; column 4, lines 37-40). Kenyon further discloses that the interface between the two liquids is constantly changed, thereby increasing the rate of transfer of a component between two liquids (column 2, lines 50-53).

90. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by contacting the sulfuric acid solution and hydrocarbon feedstream by using a fiber film contactor. One would have been motivated to do this because fiber film contactors promote efficient mass transfer between two contacting liquids.

91. Plummer discloses a process for improving the properties of aromatic concentrates comprising:

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- c) Contacting the concentrate with a sulfuric acid solution and subsequently hydrotreating the acid treated product with a catalyst containing Groups VI-B and VIII metal oxides (column 3, lines 13-20). Plummer further discloses that the hydrotreatment is effective in improving the properties, such as color and odor, of the aromatic concentrates (column 2, lines 44-45).

92. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by hydrotreating the acid treated product with a catalyst containing Group VI and VIII metal oxides in order to effectively remove sulfur contaminants and provide a higher quality product.

93. Regarding claim 25, Skomoroski discloses that the feedstream contains 0.5-4.0 wt.% nitrogen (500-4000 wppm), which encompasses the claimed range of 75-800 wppm nitrogen (column 2, line 49).

94. Regarding claim 26, Skomoroski discloses that the nitrogen present in the feedstream includes aromatic nitrogen compounds. Skomoroski does not expressly disclose that the feedstream contains carbazole and/or substituted carbazole.

However, as evidenced by Min et al ("Carbazole-type compounds in crude oils"), nitrogen-containing species in petroleum consist of carbazole-type compounds, such as carbazoles and their derivatives (benzocarbazoles, dibenzocarbazoles and their alkylated derivatives) (Chinese Science Bulletin, Vol. 43 No. 8, p. 669). Therefore, the feedstream disclosed by Skomoroski would inherently contain carbazole and/or substituted carbazole compounds.

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95. Regarding claim 27, Plummer discloses acid treating with sulfuric acid containing at least about 85% by weight  $\text{H}_2\text{SO}_4$  (column 3, lines 21-23). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski in view of Plummer and in view of Kenyon by using a sulfuric acid solution containing about 85% by weight  $\text{H}_2\text{SO}_4$ , as suggested by Plummer. One would have been motivated to do this in order to minimize the amount of sulfuric acid required for the process but maintain effectiveness in removing nitrogen compounds from the hydrocarbon feedstream.

96. Regarding claim 30, Skomoroski discloses an example in which the feedstream initially contained 2.07 wt.% nitrogen (2070 wppm) and the process removed over 90% of the nitrogenous contaminants, corresponding to a final concentration of nitrogen in the product stream of less than about 100 wppm (column 2, lines 54-55 and 67-68).

97. Regarding claim 31, Skomoroski discloses that the sulfur content of a feedstock with 0.4 wt.% sulfur is reduced to a negligible amount, corresponding to a product with a sulfur content that is at least 0.1 wt.% to approximately 0.4 wt.% less than the feedstream (column 4, lines 11 and 16-17).

98. Regarding claim 35, Plummer discloses separating the acid solution from the product after acid treatment, thereby substantially removing the sulfuric acid from the feed material (column 3, lines 30-31). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski in view of Plummer and Kenyon by separating the sulfuric acid



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from the product, as suggested by Plummer. One would have been motivated to do this in order to provide a product which is substantially free of the acid solution.

99. Regarding claim 38, Skomoroski discloses contacting the product with caustic and water; however, this step occurs after hydrotreatment (column 2, lines 12-21).

100. Plummer discloses contacting the product with caustic prior to hydrotreating. The caustic solution is added in an amount sufficient to neutralize the acid values remaining in the product, or in other words, reduce the total acid number (column 3, lines 32-35; column 6, lines 22-24). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski in view of Plummer and Kenyon by doing the caustic contacting stage prior to hydrotreatment in order to neutralize the hydrocarbon product and more readily prepare it for hydrotreatment.

**101. Claims 28, 29 and 32 are rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Plummer et al (U.S. Patent 3,487,012) and in view of Kenyon (U.S. Patent 3,758,404) and in further view of Lockwood et al (U.S. Patent 3,749,666).**

102. Skomoroski in view of Plummer and Kenyon is relied upon as set forth above in the rejection of claims 24, 27 and 31.

103. Regarding claims 28 and 29, Skomoroski in view of Plummer and Kenyon does not expressly disclose that the sulfuric acid solution is obtained from an alkylation process unit and further, that water is added to the solution to adjust the sulfuric acid concentration of the solution.

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104. Lockwood discloses a method for the improvement of petroleum distillate comprising contacting (emulsifying) the petroleum stream with spent alkylation acid (column 2, lines 2-8). Lockwood further discloses that while fresh sulfuric acid can be used, there is an economic advantage to using spent alkylation acid (column 2, lines 45-48). Lockwood teaches that the alkylation acid contains water as obtained from the alkylation unit and that controlling the acidity of the acid treating solution minimizes oil loss from the petroleum feed (column 2, lines 40-42; column 3, lines 7-9).

105. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski in view of Plummer and Kenyon by using spent sulfuric acid from an alkylation process unit in order to reduce the amount of fresh sulfuric acid required for the process. It further would have been obvious to use water to adjust the acidity, or concentration or sulfuric acid in the solution, in order to minimize oil loss from the petroleum feed.

106. Regarding claim 32, Skomoroski in view of Plummer and Kenyon does not expressly disclose that the yield loss attributed to the sulfuric acid treatment is about 0.5 to about 6 wt.%.

107. Lockwood discloses that controlled acidity minimizes oil loss from the petroleum distillate feed (column 3, lines 7-9). Therefore, it would have been obvious to a person of ordinary skill in the art at the time the invention was made to control the acidity of the acid solution and hydrocarbon feedstream contacting stage in order to minimize loss associated with the sulfuric acid.

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**108. Claims 36 and 37 are rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Plummer et al (U.S. Patent 3,487,012) and in view of Kenyon (U.S. Patent 3,758,404), and in further view of Norman (U.S. Patent 4,432,865).**

109. Skomoroski in view of Plummer and Kenyon is relied upon as set forth above in the rejection of claims 24 and 35.

110. Regarding claims 36 and 37, Skomoroski in view of Plummer and Kenyon does not expressly disclose the separation device used to separate the sulfuric acid solution from the product.

111. Norman discloses a process for treating used motor oil or synthetic crude oil with a mineral acid, such as sulfuric acid, for removing undesired nitrogen-containing materials (column 1, lines 18-21; column 8, line 3). Norman discloses that the acid solution is separated from the oil product by high speed centrifuges or clay or cellulose fiber films and the residue from the separation contains the metal contaminants and acid solution (component A) (column 14, lines 11-18).

112. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski in view of Plummer and Kenyon by using high speed centrifuges or fiber films to separate the sulfuric acid solution from the product in order to effectively remove the acid solution and contaminants from the desired product.

**113. Claims 39 and 40 are rejected under 35 U.S.C. 103(a) as being unpatentable over Skomoroski et al (U.S. Patent 3,123,550) in view of Parvinen (U.S. Patent**

**6,007,722) and in view of Kenyon (U.S. Patent 3,758,404) and in view of Chellis et al (U.S. Patent 2,984,617).**

114. Regarding claims 39 and 40, Skomoroski discloses a method for hydrotreating a shale oil distillate feedstream containing both nitrogen and sulfur contaminants (column 1, lines 10-17; Column 2, Table 1). The process comprises:

- d) Contacting the feedstream containing both nitrogen and sulfur heteroatoms with a sulfuric acid solution under conditions effective at removing greater than about 90% of the nitrogen compounds contained in the feedstream, thereby producing at least a product and a used sulfuric acid solution, wherein the volumetric treat rate of the sulfuric acid solution is in the range of about 0.5 vol.% to about 5 vol.%, based on the feedstream (column 3, lines 53-67; Fig. 1); and
- g) Hydrotreating the diesel boiling range product with a hydrotreating catalyst containing cobalt or nickel (Group VI metals) under conditions effective at removing or converting at least a portion of the sulfur contained in the product. (column 3, lines 20-24 and 59-61; column 4, lines 11-12 and 16-17).

115. Skomoroski discloses that the shale oil distillate feedstream boils in the range of 350-1000°F (column 2, line 46), encompassing a diesel boiling range feedstream boiling in the range as claimed. Skomoroski further discloses contacting the product with caustic and water; however, this step occurs after hydrotreatment (column 2, lines 12-21).

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116. Skomoroski does not expressly disclose:

- a) Combining and olefinic hydrocarbon feedstream containing C<sub>4</sub> olefins with isobutane to form a hydrocarbonaceous mixture;
- b) Contacting the hydrocarbonaceous mixture with sulfuric acid under conditions effective for producing at least an alkylate and a sulfuric acid solution having an acid concentration of at least about 75 wt. %.
- c) Adding water to the sulfuric acid solution to adjust the sulfuric acid concentration to about 75 wt.% to about 88 wt.%, based on the sulfuric acid solution;
- d) Contacting by a non-dispersive method selected from packed beds of inert materials and fiber film contactors wherein the yield loss attributed to the sulfuric acid solution is about 0.5 to about 3 wt.%;
- e) Separating the product and used sulfuric acid solution through the use of a separation device selected from settling tanks or drums, coalescers, electrostatic precipitators, and other similar devices;
- g) hydrotreating with a catalyst containing at least one Group VI metal oxide and at least one Group VIII metal oxide.

117. Parvinen discloses a process for recycling and reusing spent sulfuric acid obtained from an alkylation process (column 1, lines 60-64; column 2, lines 23-25). The alkylation process comprises:

- a) Combining an olefinic hydrocarbon feedstream containing C<sub>4</sub> olefins (butene) with isobutane to form a hydrocarbonaceous mixture;

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- b) Contacting the hydrocarbonaceous mixture with sulfuric acid under conditions effective for producing at least an alkylate and a sulfuric acid solution having an acid concentration of at least about 75 wt. % (85-88%) (column 1, lines 57-64; column 2, lines 5-7); and
- c) Adding water to the sulfuric acid solution to adjust the sulfuric acid concentration (column 3, lines 32-33).

118. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by obtaining the sulfuric acid from an alkylation process and using water to adjust the sulfuric acid concentration in the solution, as suggested by Parvinen.

119. One having ordinary skill in the art would have been motivated to do this in order to produce alkylates and efficiently use sulfuric acid for multiple processes, reducing the amount of fresh sulfuric acid required for the process.

120. Kenyon discloses a process for liquid-liquid mass transfer comprising:

- d) Contacting a hydrocarbon feedstream with a sulfuric acid solution through the use of a fiber film contactor (column 2, lines 20-30; column 4, lines 37-40); and
- e) Separating the product and sulfuric acid solution through the use of a fiber film contactor (column 2, lines 55-58).

121. Kenyon further discloses that the interface between the two liquids is constantly changed, thereby increasing the rate of transfer of a component between two liquids (column 2, lines 50-53).

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122. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by contacting the sulfuric acid solution and hydrocarbon feedstream by using a fiber film contactor and subsequently separating the product and used sulfuric acid with the fiber film contactor.

One would have been motivated to do this because fiber film contactors promote efficient mass transfer between two contacting liquids, such as a hydrocarbon stream and sulfuric acid solution, and are also effective in separating the two liquids.

123. Chellis discloses a hydroprocessing process for hydrocarbon feedstreams containing nitrogen and sulfur contaminants comprising:

- d) Contacting a sulfuric acid solution with the hydrocarbon feedstream such that the yield of the hydrocarbon product from acid denitrogenation is 98-99% of the charge volume, corresponding to a yield loss of 1-2 vol. % (column 4, line 62).
- g) Hydrotreating with a cobalt oxide-molybdenum oxide catalyst to reduce the sulfur content (column 1, lines 28-31).

124. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify the process as disclosed by Skomoroski by minimizing the loss of hydrocarbons in order to ensure a high product yield. Further, it would have been obvious to hydrotreat the product with a cobalt oxide-molybdenum oxide catalyst in order to decrease the sulfur content in the product.

***Conclusion***

125. Any inquiry concerning this communication or earlier communications from the examiner should be directed to RENEE ROBINSON whose telephone number is (571)270-7371. The examiner can normally be reached on Monday through Thursday 7:30-5:00.

126. If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Walter Griffin can be reached on (571)272-1447. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

127. Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/R. R./  
Examiner, Art Unit 1797  
9 February 2009

/Walter D. Griffin/  
Supervisory Patent Examiner, Art  
Unit 1797